Earth Surface Processes and Landforms
Earth Surf. Process. Landforms **23**, 663–668 (1998)

SHORT COMMUNICATION

ON STANDARDIZING THE ‘WATER DROP PENETRATION TIME’ AND THE ‘MOLARITY OF AN ETHANOL DROPLET’ TECHNIQUES TO CLASSIFY SOIL HYDRPHOBICITY: A CASE STUDY USING MEDIUM TEXTURED SOILS

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Received 21 January 1998; Revised 1 April 1998; Accepted 2 April 1998

ABSTRACT

Two common methods to assess soil hydrophobicity are the ‘Water Drop Penetration Time’ and ‘Molarity of an Ethanol Droplet’ techniques. For these, uncertainty exists regarding the representativeness of laboratory tests reflecting field conditions, their replicability, and the comparability of results between the two techniques. Using air-dried soils with a broad particle size and hydrophobicity range, this study shows that a high representativeness and replicability of results can be achieved. A close relationship between the two tests was found for highly, but not for moderately hydrophobic soils. Guidelines are suggested to increase representativeness, replicability and comparability of results in future studies. © 1998 John Wiley & Sons, Ltd.

KEY WORDS: soil hydrophobicity; water repellency; water drop penetration time; molarity of an ethanol droplet.

INTRODUCTION

Wettability measurements have long been used for petroleum or textile engineering purposes, with industrial standards being established for some of them (Norris, 1963; Anderson, 1986). The methods employed to measure hydrophobicity (water repellency) of soils are fewer and less standardized. Of these only some are well suited for large sample sizes, amongst which the ‘Water Drop Penetration Time’ (WDPT; Letey, 1969) and the ‘Molarity of an Ethanol Droplet’ (MED; also known as ‘Percentage Alcohol Test; Watson and Letey, 1970; King 1981) techniques have become the two most commonly used in the last two decades. The WDPT test measures how long hydrophobicity persists on a porous surface and it thus relates to the hydrological implications of hydrophobicity because the amount of surface runoff is affected by the time required for the infiltration of raindrops (Wessel, 1988). This test has been widely used especially in the laboratory (e.g. Roberts and Carbon, 1971; Ma’shum and Farmer, 1985; Brock and DeBano, 1991; Dekker and Ritsema, 1994), although it is not clear how well measurements on disturbed and bulked soil samples reflect the actual hydrophobicity of an *in situ* soil surface in the field. Furthermore, Crockford *et al*. (1991) reported that reproducibility of results was poor.

The MED test is an indirect measure of the surface tension of the soil surface and indicates how strongly a water drop is repelled by a soil at the time of application (i.e. how strongly it will ‘ball up’). This is closely related to persistence time of hydrophobicity (WDPT) according to Crockford *et al*. (1991) and Harper and Gilkes (1994), but not according to Dekker and Ritsema (1994). An advantage of the MED test is its speed so that it is well suited to field investigations where long persistence times make the WDPT technique impossibly laborious (Wallis *et al*., 1991).

As both techniques have been employed using different standards and classifications, comparison of results within and between studies is often difficult. Furthermore, most studies have been conducted on texturally

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CCC 0197-9337/97/070663–06 $17.50

Table I. Threshold WDPT times (in seconds) used by various authors to classify soils into different repellency classes

Classification	Adams *et al.* (1969)	Bisdom *et al.* (1993)	Doerr *et al.* (1996)	Ma'shum & Farmer (1985)	McGhie & Posner (1981)	Roberts & Carbon (1971)
Hydrophilic	<10	<5	<60	<1	<60	<1
Slightly hydrophobic	10–60	5–60	–	–	–	1–10
Strongly hydrophobic	–	60–600	–	–	–	10–60
Severely hydrophobic	>60	600–3600	–	–	–	>60
Extremely hydrophobic	–	>3600	>3600	–	–	–

Table II. WDPT categories used in this study

Category (time in seconds)										
<5	5–10	10–30	30–60	60–180	180–300	300–600	600–900	900–3600	3600–18 000	>18 000

relatively homogeneous sandy soils. This study uses sandy loam soils comprising a broad range in particle size and degree of hydrophobicity to (a) assess how well measurements taken on air-dried and sieved laboratory samples reflect the hydrophobicity of an undisturbed, dry *in situ* soil surface in the field; (b) examine the reproducibility of hydrophobicity results in the laboratory; and (c) determine the comparability of MED and WDPT test results. Based on these results, standards for the MED and WDPT techniques are suggested.

METHODS AND MATERIALS

Water Drop Penetration Time test

The WDPT test involves placing a water drop on a soil surface and recording the time taken for its complete penetration (Letey, 1969). If hydrophobicity were always persistent (i.e. independent of the duration of contact with water) a water droplet would penetrate a porous material either instantly (hydrophilic soil) or remain on the surface until it evaporated (hydrophobic soil) (Tschapek, 1994). Thus, if the water droplet does not penetrate instantly, it indicates that the surface tension of the soil surface is below that of the droplet (72.75 dynes cm^{-1} at 20°C). However, a decay of hydrophobicity can usually be observed in soils, and thus, measuring the delay in droplet infiltration reflects the time the surface tension of the soil remains higher than that of the droplet (i.e. how long hydrophobicity persists). WDPT can vary from instant penetration to many hours and numerous classifications exist relating WDPT categories to severity levels in hydrophobicity (Table I). These classification times are essentially arbitrary; however, they are useful in summarizing and comparing different levels of hydrophobicity. To cover all possible WDPTs, the test would have to be extended until all droplets had infiltrated. This can become very time-consuming and ultimately evaporation of droplets will influence results for very hydrophobic samples. In practice, in many studies the WDPT tests have been terminated after several minutes.

In this study, the WDPT test was standardized as follows. According to the type of experiment five or 15 drops of distilled water (20°C) were applied to the soil surface using a hypodermic syringe. The penetration time category of each drop was recorded and the median penetration time taken as representative of the WDPT of each sample. Rather than using only the five categories presented in Table I, 11 categories were used here to allow a higher resolution of WDPTs (Table II). Samples with a WDPT in excess of 1 h were carefully covered with lids to prevent drop evaporation, allowing the tests to be extended to >5 h.

The Molarity of an Ethanol Droplet test

The MED test uses the known surface tensions of standardized solutions of ethanol in water. Drops of those dilutions are applied to a soil surface and their instant or short-term (several seconds) infiltration behaviour is observed (Watson and Letey, 1971). A droplet with a higher surface tension than that of the soil surface will remain on it for some time, whereas a droplet with a lower surface tension will infiltrate instantly. Applying drops with increasing surface tensions (decreasing ethanol concentrations) until a drop resists infiltration, allows the classification of the soil into a surface tension category between two ethanol concentrations. If small concentration increments are used, the surface tension at the time of initial contact can be determined quite

Table III. Typical particle size range for soils used in this study

Size (mm)	2–1	1–0·5	0·5–0·25	0·25–0·125	0·125–0·063	0·063–0·002	<0·002
Weight (%)	16·6	7·9	4·4	7·2	13·1	45·2	5·6

accurately. The time that is allowed for the drop to infiltrate varies between authors. King (1981) used 5 s, Crockford *et al*. (1991) 3 s, whereas Harper & Gilkes (1994) used 10 s.

In this study, drops (0·05 ml) of prepared solutions were applied onto the smoothed soil surface using medical droppers. Increasing ethanol concentrations (0, 3, 5, 8·5, 13, 24 and 36 per cent by volume) were used until drop penetration occurred within 3 s. This time interval was, in accordance with Crockford *et al*. (1991), found to be observed most easily. Keeping the penetration time short was considered important to avoid the possible decay of hydrophobicity affecting the results. The above concentrations covered the range of field hydrophobicity encountered and accord with some of the concentrations used elsewhere (Crockford *et al*., 1991; Dekker and Ritsema, 1994). To facilitate statistical analysis, the MED values are categorized into a simple numerical and descriptive scale, ranging from 1 (very hydrophilic) to 7 (extremely hydrophobic).

Samples and treatment

Within a wider research programme (Coelho *et al*. 1995), hydrophobicity was investigated of sandy loam Humic Cambisols and Umbric Leptosols (FAO, 1988) on slopes afforested with *Pinus pinaster* and *Eucalyptus globulus* in the Águeda river basin in north-central Portugal (40°35′N, 8°26′W). A typical soil textural composition (<2 mm) is given in Table III. Skeletal content (>2 mm) ranged from 20 to 40 volume per cent. Further details of study area and soils are given in Doerr *et al*. (1996, 1998). Hydrophobicity was measured on (a) undisturbed *in situ* soil surfaces in the field under dry conditions (summer drought) and (b) on laboratory samples from the surface (0 to 2 cm depth) and from depths of up to 30 cm. Prior to field testing, any surface organic debris was removed from the mineral soil surface. The median of five MED measurements undertaken within an area of approximately 10 cm × 10 cm was taken as being representative of the site.

For laboratory testing, soil was air-dried and carefully hand-sieved to remove the skeletal fraction (>2 mm). Approximately 10 g of soil was put into clear circular plastic dishes (50 mm diameter; 10 mm depth) and surfaces were carefully smoothed by hand. For both MED and WDPT tests, drops were emplaced from no higher than 5 mm to avoid excess kinetic energy affecting soil–droplet interaction.

Individual experiments

Reliability of laboratory testing to reflect field hydrophobicity. To examine how closely measurements taken on air-dry and sieved laboratory samples reflect the *in situ* hydrophobicity of an undisturbed field soil, MED measurements were conducted at the soil surface for 82 undisturbed sites which were subsequently sampled, air-dried and hand-sieved in the laboratory and MED tested again.

Replicability of hydrophobicity results in the laboratory. To examine the replicability of hydrophobicity test results in the laboratory, 71 air-dried and sieved samples from different depths were each split into three subsamples, each of which was then WDPT tested using 15 drops to minimize the effect of possible variations within subsamples. Fifteen was found to be about the largest number of droplets for which the penetration time could be sufficiently recorded by one observer. The replicability was assessed using the average standard deviation in a subsample and by determining the range of WDPT categories within the three subsamples for each sample. In this experiment, WDPTs were only measured to >1 h, and thus results are based on the first 10 WDPT categories of Table II.

Comparability of MED and WDPT test results. To determine the comparability of MED and WDPT tests, results are compared for 176 soil samples having both tests conducted under laboratory conditions on the same day and on the same sample. To allow both tests to be conducted within the available surface area of the same sample, five instead of the 15 drops used in the previous experiment were applied in the WDPT test allowing the MED test to be performed on soil where no droplets had previously infiltrated. This was essential as trials had shown that at the point of water droplet infiltration, hydrophobicity was not re-established after the soil was dry again. The 11 WDPT time categories given in Table II were reduced to seven to facilitate a direct comparison with the seven MED classes used in this study. Following the classification of Dekker and Ritsema (1994),

categories within 5–60 s, 3–10 min or >10 min to 1 h were amalgamated.

Table IV. Comparison of field and laboratory MED tests results (the seven classes used are explained in Table VI)

Comparison	identical	lab.<field (1 class)	lab.>field (1 class)	lab.<field (2 classes)	lab.>field (2 classes)
Occurrence (%)	35	60	4	0	1
Number (n=82)	29	49	3	0	1

Table V. Frequency distribution of ranges of median WDPT test results within three subsamples for 71 samples using 10 WDPT categories. Figures are the percentage (number) of samples with a subsample range of the size specified. The categories (<5 s to >1 h) are explained in Table II)

Range	no range*	1 category	2 categories	3 categories	>3 categories
Occurrence (%)	65	31	3	1	0
Number (*n*=71)	46	22	2	1	0

* All three in same category

Table VI. Frequency distribution of laboratory MED (ethanol concentrations and classes), corresponding WDPT categories for 176 samples and their descriptive label used in this study

Hydrophobicity (MED)			Hydrophobicity (WDPT)						
Class	Descriptive label	Ethanol %	<5 s	5–60 s	60–180 s	180–600 s	600–3600 s	1–5 h	>5 h
7	Extremely hydrophobic	36	–	–	–	–	–	–	5
6	Very strongly hydrophobic	24	–	–	–	–	8	36	48
5	Strongly hydrophobic	13	–	–	1	6	13	1	13
4	Moderately hydrophobic	8·5	–	6	7	1	–	–	–
3	Slightly hydrophobic	5	–	14	2	–	9	–	–
2	Hydrophilic	3	–	–	–	–	–	–	–
1	Very hydrophilic	0	6	–	–	–	–	–	–

RESULTS

Reliability of laboratory measurements to reflect field hydrophobicity

Field and laboratory MED values match relatively closely, 99 per cent being in the same or only one class above (60 per cent) or below (4 per cent) that measured in the field (Table IV).

Replicability of hydrophobicity results in the laboratory

The spread of hydrophobicity results over the 10 categories for the three subsamples is very limited with a mean standard deviation of 0·186 categories. For 96 per cent of all samples, the WDPTs of the subsamples have no range or a range of one category and only 4 per cent range over more than one category (Table V).

Comparability of MED and WDPT test results

The coefficient of correlation between the tests in 0·73 (p<0·01, n=176, Spearman Rank correlation). All MED values of Class 7 (36 per cent ethanol) corresponded to WDPTs of >5 h and 90 per cent of all MED Class 6 (24 per cent ethanol) values corresponded to WDPTs > 1 h. Thus, the highest MED values were found to be associated with the highest WDPT categories. However, MED values do not relate consistently to certain WDPT categories as for example WDPTs range from 60 s to >5 h for samples within MED Class 5 (13 per cent ethanol) (Table VI).

DISCUSSION

Representativeness of laboratory testing to reflect hydrophobicity

Field sampling and laboratory procedures are thought to affect hydrophobicity values in several ways. For example, Roberts and Carbon (1971) found that the upper few millimetres of a mineral soil were often

hydrophilic and Crockford *et al*. (1991) reported that mixing the top 1·5 cm of a soil greatly reduced hydrophobicity. Thus, the value measured on a surface sample in the laboratory, even if the soil has been sampled to a depth of less than 2 cm, might not accurately reflect the hydrophobicity of the *in situ* soil surface. Also, drying procedures could influence hydrophobicity. Ma'shum and Farmer (1985) and Franco *et al*. (1995) found that oven-drying of moist samples resulted in much higher levels of hydrophobicity than air-drying. Furthermore, sieving could affect the results as hydrophobic skins might be removed from soil particles by abrasion (Ma'shum and Farmer, 1985; Wallis *et al*., 1991) and particles >2 mm are removed.

The present results indicate that for mixed particle size range soils, laboratory measurements on air-dried and carefully hand-sieved samples broadly reflect the hydrophobicity of a dry, undisturbed *in situ* soil surface with a tendency to slightly underestimate field hydrophobicity (Table IV). A reasonable correspondence of field and laboratory results has also been found for soil at greater depths in the study area (Doerr *et al*., 1996). There is good reason to believe, therefore, that the sampling and laboratory procedure adopted here can give a good measure of *in situ* hydrophobicity even for soils with a mixed particle size. This might, however, only apply to soils in which hydrophobicity is relatively consistent with depth as is the case in the study area. Where hydrophobicity varies within a few centimetres, a close correspondence of field and laboratory values may not be given. Thus, some field tests under dry conditions should always be included to assess to what extent the destruction of the original soil surface and the inevitable mixing of material affects the results.

Replicability of hydrophobicity results in the laboratory

Poor replicability, as experienced by Crockford *et al*. (1991), and thus poor reliability of hydrophobicity test results might be expected, particularly in samples with a broad particle size range, because hydrophobicity can vary with particle size fraction (Crockford *et al*., 1991; Doerr *et al*., 1996). The results of this study indicate that for laboratory testing of sieved and thus thoroughly mixed samples, a low replicate variability can be achieved. Therefore, for WDPT testing, the procedures used in this study, including the application of a small number of drops (three to five), can be recommended.

Comparability of MED and WDPT test results

Most hydrophobicity studies have been based on one of the two tests or the MED test was used in the field and the WDPT test in the laboratory (e.g. Crockford *et al*., 1991) and comparison of test results is often difficult. In Crockford *et al*.'s study, a close but unquantified relationship of the test results was observed for most, but not all samples. In another Australian study, Harper and Gilkes (1994) found a good correlation between both methods for WDPTs ranging from 10 to 200 s and computed a conversion formula ($\mathrm{MED} = -1{\cdot}3 + 1{\cdot}1 \log \mathrm{WDPT}$). However, in an extensive study on Dutch sand dunes, Dekker and Ritsema (1994) found only a limited correlation between test results over a much broader range of hydrophobicity levels (<5 s to >6 h). As in this study, for low MED values (5 to 8 per cent), corresponding WDPTs ranged from 60 s to >1 h, whereas ethanol concentrations above 24 per cent were found mainly to represent a WDPT of >1 h, Thus, not only for relatively homogeneous coarse sandy soils, but also for soils with a wide range of particle sizes as used in this study, long persistence times of hydrophobicity (WDPTs>1 h) seem to be associated with a high degree of hydrophobicity (MED). Contrasting Crockford *et al*. (1991) and Harper and Gilkes (1994), however, a very close correspondence of WDPTs with MED values seems not to be given for samples with a WDPT between 60 s and 1 h. It is therefore recommended here that if a wider comparability of results is envisaged or one test is used in the field and the other in the laboratory, both tests should also be conducted under the same conditions on a range of selected samples to provide a measure of comparability of results.

CONCLUSION

This study has shown that for the soils and procedures chosen in this study (a) field hydrophobicity results are matched closely by laboratory tests, (b) laboratory WDPT test results are highly reproducible, and (c) MED test results correspond reasonably well to WDPT results for highly, but less well for moderately hydrophobic soils.

The soils used in this study comprise a broad hydrophobicity and particle size spectrum and categories used to classify hydrophobicity allow a comparatively detailed discrimination of a wide range of degrees of water

repellency. It is thought, therefore, that in order to standardize hydrophobicity measurements on dry soils and improve reliability and comparability of results within and between studies, the sample preparation and measurement techniques used here can be recommended for sandy or silty soils. The close correspondence between field and laboratory results found in this study, however, may not be matched in studies where field hydrophobicity is more variable with depth (e.g. present in thin layers). Therefore, in laboratory testing an assessment should be made of the extent to which laboratory results reflect field hydrophobicity. Soil samples should be air-dried rather than oven-dried in order to avoid the possibility of enhancing their degree of hydrophobicity. Light hand-sieving homogenizes the soil matrix aiding representativeness of the measurements. Owing to its direct hydrological relevance, the WDPT test may be more appropriate to measure and classify soil hydrophobicity, but, for soils with long penetration times, the MED test is more practical. Whichever test is selected, the inclusion of results of both tests conducted on selected samples in any study would allow a wider comparabiliy of results.

ACKNOWLEDGEMENTS

I would like to thank M. Clarke for assistance with field and laboratory measurements and E. M. Bridges and R. A. Shakesby for comments on different versions of the manuscript.

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